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JAPANESE INDUSTRIAL STANDARD

**Automatic Analysers for Sulphur in
Crude Oil and Petroleum Products**

JIS B 7995—1984

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Automatic Analysers for Sulphur in Crude Oil
and Petroleum Products

B 7995-1984

1. Scope

This Japanese Industrial Standard specifies the automatic analysers to measure the sulphur contained in crude oil and petroleum products by the use of radiation, hereinafter referred to as the "analysers".

- Remarks 1. In this standard, the "crude oil and petroleum products" means the crude oil, gas oil, and fuel oil containing little volatile matter and 0.01 to 4 mass % sulphur.
2. The method of using tabletop type analysers shall be in accordance with the testing methods for sulphur of radiation system specified in JIS K 2541. Further, though the method of using stationary type analysers shall comply with the handling instructions for the respective analysers, when the measured values are intended for quality certification, they shall have precision within that specified by the radiation system testing methods for sulphur in JIS K 2541.
3. The radiation to be used for the analysers shall be handled in accordance with relevant laws and regulations.

2. Definitions

The principal terms used in this standard, except for the following, shall comply with JIS K 0050, JIS Z 4001, JIS Z 8103 and JIS Z 8202.

- (1) zero-calibration liquid The liquid to be used in calibrating the minimum scale value (the indicating value for 0.00 mass % sulphur content) of the analyser.
- (2) span-calibration liquid The liquid to be used in calibrating the maximum scale value (to be 4 mass % sulphur content, for a tabletop type analyser) of the analyser.
- (3) calibration liquid The liquid for inspecting an indication value for sulphur content near the midpoint of scale range or the indication value for an arbitrary sulphur content of the analyser.
- (4) ratio of carbon to hydrogen (hereinafter expressed by C/H) The mass ratio of carbon to hydrogen in crude oil and petroleum products.
- (5) C/H-testing liquid Concerning the excitation system analyser, the testing liquid for testing the effect of C/H of the sample on measured values.

Applicable Standards: See page 21.

- (6) error by cell Concerning the tabletop type analyser, the error due to the difference in thickness of the cell-window material.
- (7) response-testing liquid Concerning the stationary type analyser, the testing liquid for measuring the response time.
- (8) heavy-metal-testing liquid Concerning the transmission system analyser, the testing liquid for testing the effect of heavy metals in the sample on measured values, and iron is used as the heavy metal.
- (9) parallel measurement error Concerning testing, the error in the measurement for which all of the person, date and time and analyser are the same.

3. Classification of Analysers

The analysers shall be classified by the measuring principle and type into the three kinds as shown in Table 1.

Table 1 Classification of Analysers

Classification by measuring principle	Classification by type
Excitation system	Tabletop type ⁽¹⁾
	Stationary type ⁽²⁾
Transmission system	Stationary type ⁽²⁾

Notes ⁽¹⁾ Tabletop type analysers are mainly installed in laboratories, and are used for measuring various samples intermittently.

⁽²⁾ Stationary type analysers are used for carrying out continuous measurement.

4. Excitation System Analysers

4.1 Measuring Principle The excitation system determines the sulphur contained in the sample by measuring the intensity of fluorescent X-rays at a level of about 2.3 keV that is generated by sulphur atoms when the sample is irradiated by the radiation emitted from an X-ray tube or a radioactive isotope.

4.2 Excitation System Tabletop Type Analysers

4.2.1 Performance The performance of excitation system tabletop type analysers shall comply with the following. The analysers shall have a function appropriate to the intermittent measurement of sulphur contained in crude oil and petroleum products, and shall meet the specifications of Table 2 when tested in accordance with 4.2.3.

Table 2. Performance of Excitation System
Tabletop Type Analysers

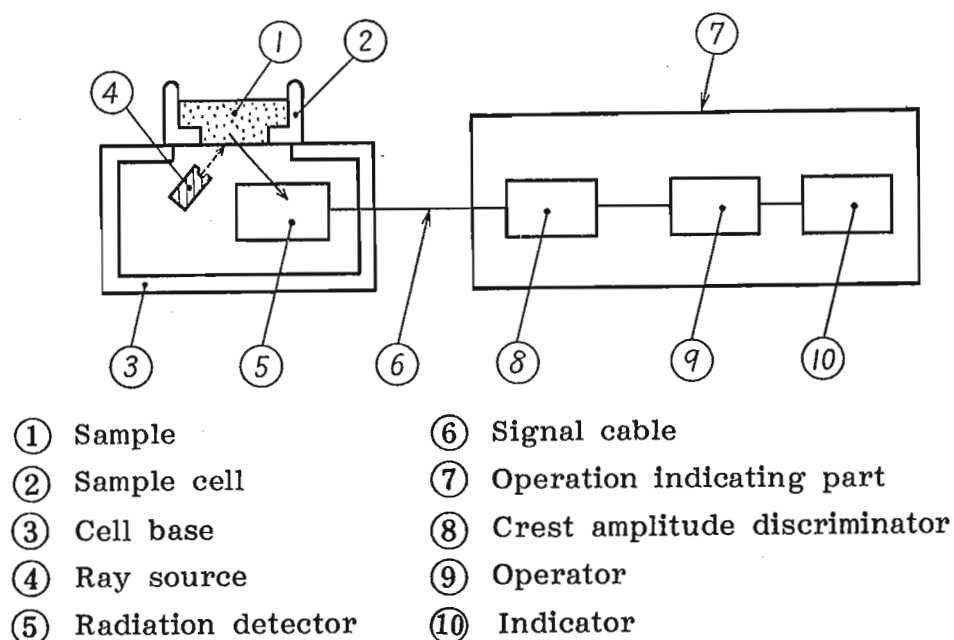
Item	Performance
Error in parallel measurement	Sulphur content: 0.01 mass % + 0.01 S, max.
Zero-drift	Sulphur content: 0.01 mass %, max.
Span-drift	Sulphur content: 0.01 mass % + 0.015 S, max.
Error in linearity of calibration line	Sulphur content: 0.02 mass % + 0.05 S, max.
Error by cell	Sulphur content: 0.02 mass % + 0.02 S, max.
Variation of measured value at 10 % variation of power source voltage	Sulphur content: 0.01 mass % + 0.01 S, max.
Variation of measured value at 5°C variation of ambient temperature	Sulphur content: 0.01 mass % + 0.015 S, max.
Variation of measured value at 1.0 variation of C/H	Sulphur content: 0.01 mass % + 0.02 S, max.
Withstand voltage	No abnormality shall occur.
Insulation resistance	5 MΩ min.

Remark: "S" stands for the sulphur content (mass %) of the calibration liquid and testing liquid used in the test.

4.2.2 Constitution of Analysers The constitution of the excitation system tabletop type analysers shall comply with the following. Furthermore, the analysers shall be constituted of a ray source, sample cell, radiation detector, operation indicating part, etc., and Fig. 1 shows an example of the constitution.

Remark: The analysers shall, in ordinary working condition, not likely to cause danger, have enough durability, be correct in shape, good in assembly, and not easily cause mechanical and electrical failure.

Fig. 1. Example of Constitution of Excitation System
Tabletop Type Analyser



- (1) Ray Source It shall be a sealed radioactive isotope or X-ray tube and radiate a radiation suitable to measurement, and in the state where the ray source is normally equipped, the leakage of radiation shall be not more than the dose prescribed in relevant laws and regulations.
- (2) Sample Cell The sample cell used shall be made of a material resistive to petroleum and solvent, and for its window, a material having little radiation-absorbing power, e.g., mica, polyester ⁽³⁾ and metallic beryllium, shall be used.

Note ⁽³⁾ It shall be not more than 10 μm in thickness and not more than $\pm 15\%$ in variation of thickness by different sample cell.

Remark: The cell that handles a volatile sample shall have a construction capable of preventing the evaporation of the sample and taking countermeasures against the deformation of window by vapour pressure.

- (3) Radiation Detector The radiation detector is a device for transducing the incident radiation into electric signal, and in general, a proportional counter tube is used.

Remark: The proportional counter tube shall be such one that generates a pulse signal with a crest value proportional to the energy of the incident radiation in proportion to its intensity.

- (4) Operation Indicating Part The operation indicating part shall be capable of treating the signal from the radiation detector, and of indicating or recording the sulphur content in a figure with not less than three decimal places.

4.2.3 Performance Test Method The performance test method of excitation system tabletop type analysers shall comply with the following.

- (1) Test Condition When test conditions are not specified in (3) Test Methods, tests shall be carried out under the following test conditions.
- (a) Ambient Temperature It shall be an arbitrary temperature between 10 and 30°C. However, it shall not vary beyond $\pm 2^\circ\text{C}$ during testing.
 - (b) Humidity It shall be an arbitrary relative humidity between 45 % and 85 %.
 - (c) Variation of Atmospheric Pressure The variation range of atmospheric pressure shall be not more than 20 mbar ⁽⁴⁾.
- Note ⁽⁴⁾ 1 bar = 10^5 Pa
- (d) Power Source Voltage It shall be the rated voltage ± 2 %.
 - (e) Power Source Frequency It shall be the rated frequency ± 0.2 Hz.
 - (f) Warming-up Time It shall be not less than 1 h.
 - (g) Determination of Measured Value One reading value (a digital or printed value) is taken as the measured value.
- (2) Testing Liquids and Others
- (a) C/H-Testing Liquid It shall be a liquid that has been prepared in accordance with Appendix.
 - (b) Zero-Calibration Liquid, Span-Calibration Liquid and Calibration Liquid They shall be the gas oil or fuel oil containing 0.00 mass %, 4 mass % and arbitrary sulphur, respectively ⁽⁵⁾, or the liquids that have been prepared in accordance with Appendix.

Note ⁽⁵⁾ The determination of sulphur contents shall be in accordance with the air method in the combustion tube system testing method for sulphur specified in JIS K 2541.

Reference: The standard sulphur content samples authorized by Japan Petroleum Institute (a corporate juridical person) for the standard values of sulphur contents as currently supplied may be used.

- (3) Test Methods The methods of testing the analysers shall comply with the following:

Furthur, preceding each test, the analyser shall be calibrated for the zero-indication value and span-indication value by the method stated in the handling instructions for the analyser with the use of zero-calibration liquid and span-calibration liquid. However, those calibrating operations may be omitted when the tests of (i) and (j) are carried out.

- (a) Test for Error in Parallel Measurement A prescribed quantity of the calibration liquid is put into one sample cell, and the sulphur content is measured successively 10 times. The difference between the maximum and the minimum values of these 10 measured values is taken as the error in parallel measurement.
- (b) Zero-Drift Test A prescribed quantity of the zero-calibration liquid is put into one sample cell, the sulphur content is measured successively 10 times, and the measured values are averaged. Thereafter, a similar measurement is repeated with the same cell over 4 h nearly at every 1 h, and five mean values are taken. The difference between the maximum and the minimum of these mean values is taken as the zero-drift.
- (c) Span-Drift Test A prescribed quantity of the span-calibration liquid is put into one sample cell, the sulphur content is measured successively 10 times, and the measured values are averaged. Thereafter, a similar measurement is repeated with the same cell over 4 h nearly at every 1 h, and five mean values are taken. The difference between the maximum and the minimum of these mean values is taken as the span-drift.
- (d) Test for Error on Linearity of Calibration Line A prescribed quantity of the calibration liquid is put into one sample cell, the sulphur content is measured successively 10 times, and the measured values are averaged. The difference between this mean value and the certified value of sulphur content of the calibration liquid is taken as the error on linearity of calibration line.
- (e) Test for Error by Different Cells A prescribed quantity of the calibration liquid is put into five sample cells, for each cell the sulphur content is measured successively 10 times, and the measured values for each cell are averaged. The difference between the maximum and the minimum of the averages for the different cells is taken as the error by different cells. In the above, the materials of cell windows are taken from the same lot.